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# III-Nitride grating grown on freestanding $\text{HfO}_2$ gratings

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## Abstract

We report here the epitaxial growth of III-nitride material on freestanding  $\text{HfO}_2$  gratings by molecular beam epitaxy. Freestanding  $\text{HfO}_2$  gratings are fabricated by combining film evaporation, electron beam lithography, and fast atom beam etching of an  $\text{HfO}_2$  film by a front-side silicon process. The 60- $\mu\text{m}$  long  $\text{HfO}_2$  grating beam can sustain the stress change during the epitaxial growth of a III-nitride material. Grating structures locally change the growth condition and vary indium composition in the InGaN/GaN quantum wells and thus, the photoluminescence spectra of epitaxial III-nitride grating are tuned. Guided mode resonances are experimentally demonstrated in fabricated III-nitride gratings, opening the possibility to achieve the interaction between the excited light and the grating structure through guided mode resonance.

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**Keywords:** InGaN/GaN QWs, fast atom beam etching, molecular beam epitaxy

## Introduction

Freestanding III-nitride structures can take advantage of the large refractive index contrast between III-nitride and air [1-7]. In such structure, the excited light has a potential to interact with freestanding structure through guided modes. Among the approaches towards creating suspended III-nitride structures, growth of III-nitride on freestanding structured template is an emerging technology. During growth process, nanoscale structures locally change the growth conditions and thus, the selective growth can be achieved to generate epitaxial III-nitride structures with smooth facets [8-11]. Meanwhile, freestanding III-nitride structures are formed by growth method and free of the etching damage. Moreover, the as-grown III-nitride structures can provide a natural optical cavity to support guided mode resonances, opening the possibility to achieve the interaction between the excited light and the epitaxial structures.

From the growth point of view, small material lattice mismatch between HfN and GaN crystals makes HfN film a superior buffer layer for the growth of GaN [12,13]. During molecular beam epitaxy (MBE) growth,

HfN surface can be formed by nitrifying  $\text{HfO}_2$  substrate. Hence, structured  $\text{HfO}_2$  film can be used as a template for growing III-nitride materials [14]. On the other hand,  $\text{HfO}_2$  film is an excellent optical material with high laser damage threshold, thermal, and chemical stability [15-17]. Recently, we have fabricated freestanding  $\text{HfO}_2$  gratings and experimentally demonstrated their guided mode resonances [18]. It is of great interest to implement the growth of III-nitride materials on freestanding  $\text{HfO}_2$  gratings.

Here, we demonstrate the freestanding III-nitride gratings grown on suspended  $\text{HfO}_2$  gratings. The epitaxial growth of InGaN/GaN quantum wells (QWs) are performed on freestanding  $\text{HfO}_2$  gratings by MBE technique. The optical performances of the resultant epitaxial structures are characterized in photoluminescence (PL) and reflectance measurements.

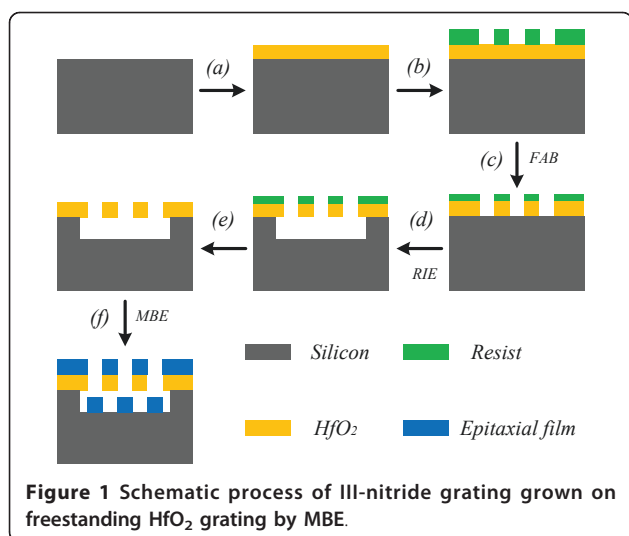
## Fabrication

The whole fabrication process is schematically illustrated in Figure 1. Freestanding  $\text{HfO}_2$  gratings are fabricated by a combination of film evaporation, electron beam lithography, fast atom beam (FAB) etching of  $\text{HfO}_2$  film with a front-side silicon process [18]. Subsequently, the epitaxial growth of III-nitride material by MBE is conducted on the suspended  $\text{HfO}_2$  gratings.

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After nitrifying HfO<sub>2</sub> substrate at the temperature of 680°C for 20 min, a 150-nm thick buffer layer is deposited at a temperature of 680°C, and a 450-nm high-temperature GaN layer is then grown at the temperature of 750°C. The six-pair 3-nm InGa<sub>0.15</sub>N/12-nm GaN MQWs is subsequently deposited at a temperature of 660°C. Finally, a 20-nm GaN layer is grown at a temperature of 660°C.

### Experimental results and discussion

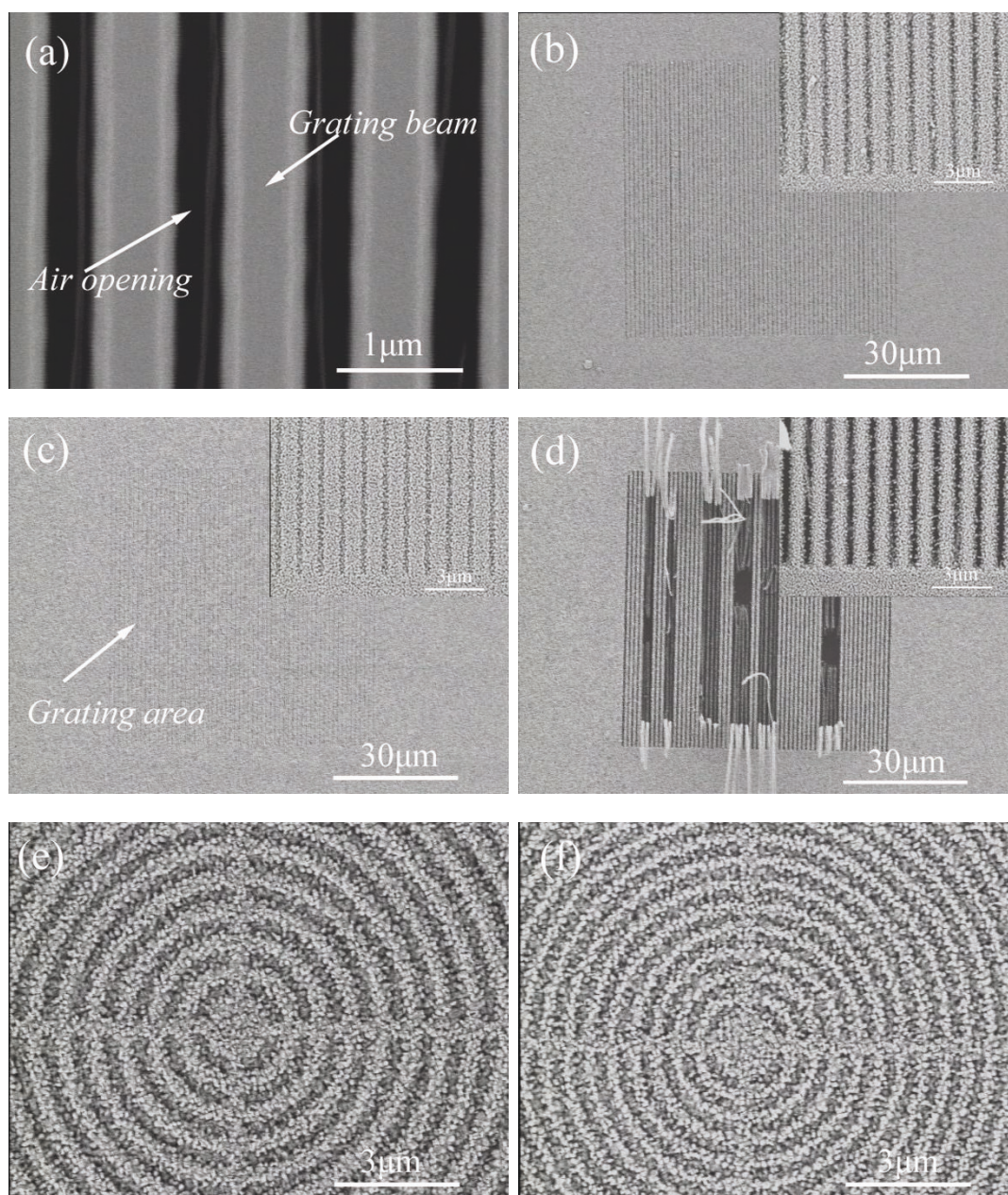
Figure 2a illustrates scanning electron microscope (SEM) images of freestanding HfO<sub>2</sub> grating template with a grating period of 1,020 nm. One period grating is comprised of HfO<sub>2</sub> grating beam and air opening. Actually, a suspended HfO<sub>2</sub> grating beam functions as a template for the epitaxial growth of III-nitride materials. Since the original HfO<sub>2</sub> grating beam has a trapezoidal profile, we use the designed air opening to describe the epitaxial III-nitride grating for simplicity. Figure 2b shows the 60-period epitaxial grating grown on 1,020 nm period suspended HfO<sub>2</sub> grating with the air opening of 400 nm, and the inset is the zoom-in image of freestanding III-nitride grating. The 60-μm long suspended HfO<sub>2</sub> grating beam can guarantee sufficient stiffness for MBE growth. III-Nitride nanocolumns are grown on the top surface and the sidewall of the grating beam. The flat III-nitride grating surface can be achieved by improving the growth condition. Decreasing the original air opening to 200 nm, the resultant grating opening illustrated in the inset of Figure 2c is easily filled with III-nitride nanocolumns, and the epitaxial III-nitride grating is similar to that grown on unpatterned area, as shown in Figure 2c. Increasing the original air opening to 600 nm, the epitaxial grating beams shown in Figure 2d are in the tendency of being deflected and fragile.

Figure 2e,f show the epitaxial circular gratings with grating period of 700 and 500 nm, respectively. It can be clearly seen that the grating openings are easily filled with III-nitride nanocolumns as the grating period decreases, especially for small period grating.

The emission properties of the resultant epitaxial structures are characterized using a micro-PL system at room temperature. The excitation source is a continuous wave 325 nm He-Cd laser source, and the pump light is focused on the sample through a UV-compatible objective lens (×20 and numerical aperture, 0.36). The emitted light is collected by the same objective lens and measured using a multichannel analyzer system (Hamamatsu C10027). Figure 3a shows the PL spectra of 1,020-nm period epitaxial gratings with various air openings. Regarding the excitation of InGa<sub>0.15</sub>N/GaN QWs grown on an unpatterned area, two distinct PL peaks are observed around 441 and 620.5 nm, and a clear broad shoulder is found at approximately 748.7 nm in the PL spectra. The emission from epitaxial materials is dependent on indium content in the InGa<sub>0.15</sub>N quantum well [19]. Variations in growth temperature leads to indium composition fluctuations and eventually results in the broad emission spectra [20–22]. Compared with unpatterned substrates, grating structures locally change the epitaxial growth conditions, giving an influence on indium composition in the epitaxial gratings. It can be seen that the tuning of the PL spectra is much more obvious as the original air opening increases. As to the grating with a designed air opening of 400 nm, only two PL peaks are observed around 437.9 and 662.6 nm, respectively. The PL intensity is also improved for epitaxial grating structures due to their freestanding characteristics. Figure 3b illustrates the PL spectra of circular epitaxial gratings versus grating period. As the grating period decreases, the measured PL spectra tend to be similar to those of unpatterned area, except a distinct increase in PL intensity of approximately a 434-nm peak.

Since the epitaxial gratings are freely suspended with air as the low refractive index material on the top and bottom, the freestanding epitaxial gratings can serve as an optical cavity to support guided mode resonances, which are characterized in reflectance measurement with the tunable laser operating in the range of 1,460 nm to approximately 1,580 nm. The optical responses of the freestanding epitaxial gratings are dependent on the polarization due to their one-dimensional configuration. The reflectance spectra versus polarization are obtained by rotating the sample with an angle of 90° with respect to the initial measurement. Under transverse electric (TE) polarization (TE is polarized in the plane of the grating and parallel to the grating lines), one sharp reflection dip approximately 4% is clearly observed at 1,500.5 nm for 1,040 nm period epitaxial grating. Measured reflectances over 70% are with a

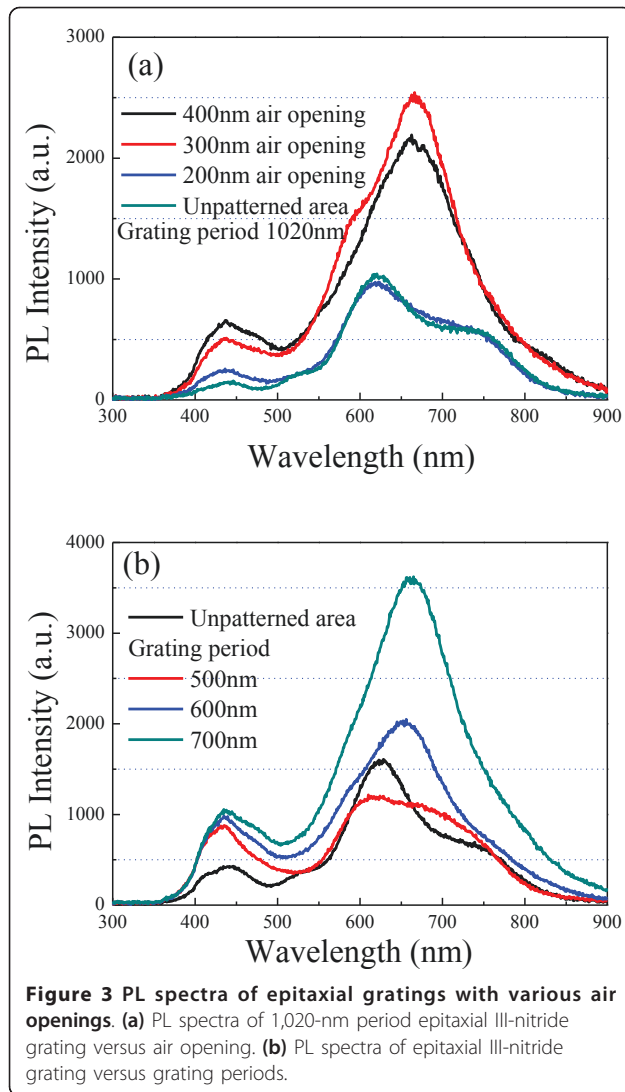




**Figure 2** SEM images of a freestanding  $\text{HfO}_2$  grating template. (a) SEM image of freestanding  $\text{HfO}_2$  grating template; (b) epitaxial III-nitride grating with air opening of 400 nm, and the inset is the zoom-in image; (c) epitaxial III-nitride grating with an air opening of 200 nm, and the inset is the zoom-in image; (d) epitaxial III-nitride grating with an air opening of 600 nm, and the inset is the zoom-in image; (e) 700-nm period epitaxial circular III-nitride grating; and (f) 500-nm period epitaxial circular III-nitride grating.

broad stopband in the range of 1,512.8 nm to approximately 1,563.5 nm. The reflection band shifts, and the shape changes under transverse magnetic polarization, which is perpendicular to the grating lines. As the grating period decreases from 1,040 to 1,020, the broad reflection band exhibits a distinct blue shift, indicating that the guided mode resonances shift proportionally to the grating period. Compared with the original  $\text{HfO}_2$  gratings, the

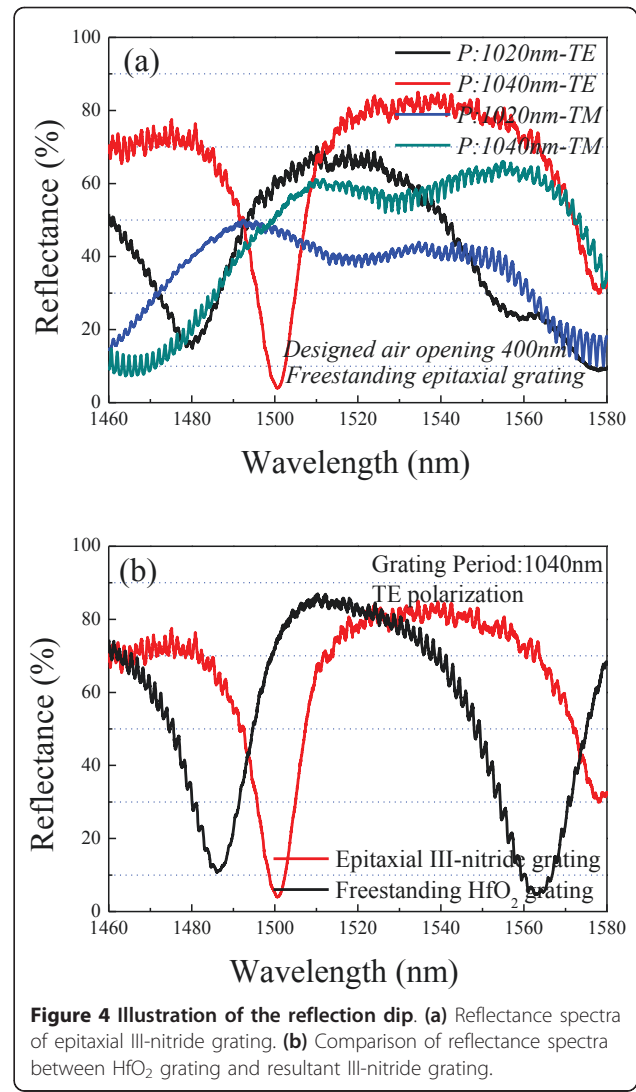
effective index of the freestanding grating is increased after the epitaxial growth of III-nitride materials, resulting in the red shifting of reflectance spectra and the broadening of the reflection band. The reflection dip illustrated in Figure 4b has a red shift of approximately 15 nm, and the reflection band widens about 10 nm. Period-, polarization-, and refractive index-dependent resonances endow freestanding III-nitride gratings the capacities to be used



for resonant optical devices. Since InGaN/GaN QWs are incorporated into the grating structures, another potential application of resonant III-nitride grating is developed for the resonant emission, where the excited light from QWs is coupled to one mode of the grating waveguide due to the match between emission wavelength and resonant wavelength.

## Conclusions

The epitaxial growth of III-nitride material is performed on the suspended  $\text{HfO}_2$  grating. The 60- $\mu\text{m}$  long free-standing  $\text{HfO}_2$  grating beam can sustain the stress change during MBE growth. The PL spectra and reflectance of epitaxial III-nitride gratings are experimentally characterized. Epitaxial III-nitride grating can function as an optical cavity to support resonance mode, which is demonstrated and compared to the resonances of original  $\text{HfO}_2$  grating. These results indicate that resonant



III-nitride gratings are promising for the development of resonant optical devices and the realization of the resonant emission. This work also opens the possibility for fabricating novel III-nitride optic devices by a combination of freestanding  $\text{HfO}_2$  nanostructures with epitaxial growth of III-nitride materials.

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## Authors' contributions

YW carried out the device design and fabrication, performed the optical measurements, and drafted the manuscript. TW carried out  $\text{HfO}_2$  film evaporation. FH conducted the epitaxial growth of III-nitride. YK participated in its design and optical characterization. HZ participated in the draft of the

manuscript and coordination. KH conceived of the study, and participated in its design and coordination. All authors read and approved the final manuscript.

#### Competing interests

The authors declare that they have no competing interests.

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